

UNIVERSITI TEKNOLOGI MARA

**PREPARATION AND
CHARACTERIZATION OF
LANTHANUM PHOSPHATE
NANOPARTICLES BY SOLGEL
SPIN COATING METHOD**

ZAINURUL ATIKAH BINTI ZAKARIA

Thesis submitted in fulfillment
of the requirement of the degree of
Master of Science

Faculty of Applied Sciences

January 2020

AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the result of my own work, unless otherwise indicated or acknowledged as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any other degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

Name of Student	: Zainurul Atikah Binti Zakaria
Student ID No.	: 2011296576
Programme	: Master in Science
Faculty	: Applied Sciences
Thesis Title	: Preparation and Characterization of Lanthanum Phosphate Nanoparticles by Solgel Spin Coating Method
Signature of Student	:
Date	: January 2020

ABSTRACT

The sol-gel method was used to prepare LaPO_4 nanoparticles as the intimate mixing of the chemical in ensuring homogeneity of the product. Besides, sol-gel methods also have much compensation compared to other methods such as easy coating of large surfaces, low processing temperature and also simple and cheap experiment set-ups. In this work, a sol-gel spin coating method was adopted to prepare LaPO_4 nanoparticles with good morphologies. $\text{NH}_4\text{H}_2\text{PO}_4$ and $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ were used as a precursor or starting material. $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8$ (EDTA) and $\text{C}_6\text{H}_8\text{O}_7$ were used as the chelating agent in the starting solution, while NH_3 solution was used as a solvent. LaPO_4 nanoparticles have been successfully prepared with different concentrations, 0.01, 0.03, 0.05, 0.08 and 0.10 mol of EDTA under constant calcinations temperature, 500°C . The morphology of LaPO_4 nanostructures deposited on glass substrate shows nano-rice, nano-sphere, and nano-particle structures. A size of sphere-like structures is smallest at concentration of EDTA 0.05 molar. The intensity of photoluminescence was found to be increased as the concentration of EDTA increased, and LaPO_4 prepared at 0.05 mol concentration of EDTA has higher intensity. This is due to highest intensity of PO_4^{3-} bonding existed. Next, the calcinations temperature to produce thin film LaPO_4 nanostructure was varied at; 200, 300, 400, and 500°C . FESEM results show that at lower annealing temperatures, 200°C and 300°C , the rice-like structures of LaPO_4 was formed and when the temperatures were increased to 400°C and 500°C , the LaPO_4 started to form a spherical shape. The intensity of photoluminescence was found to be decreased as the calcinations temperature increased and 400°C showed higher intensity compare to others. The peak shifted to the left for calcinations temperature 400°C and the shifting of the peak to the right at 500°C . Using the same sample preparation condition of 0.05 molar concentration of EDTA and 400°C calcinations temperature, the number of LaPO_4 coating layer were varied to 1, 3 and 5. Different coating on the glass will contribute to the different sizes and morphologies of the LaPO_4 nanostructures thin film. With increasing number of layers, the particles appear to stick together and this increases the density of nanoparticles formed on surface. The intensity of photoluminescence was found to be decreased as the number of layers increased and 1 layer showed the higher intensity. When the number of layers increased, the peak was shifted to the left and photon emission energy recorded was increased.

ACKNOWLEDGEMENT

Assalamualaikum w.b.t,

Firstly, I would like to express our grateful to ALLAH S.W.T for giving me the mercy and good health to finish my thesis as the way it has to be. I also would like to thank to my most helpful and respected supervisor, Prof. Dr. Saifollah bin Abdullah for his time, co-operation, support and encouragement shown to me throughout my study and a special thank you goes my co-supervisor, Prof. Dr. Mohamad Rusop bin Mahmood who had given a lot of co-operation and favor to guide me along the process in completing this project. Besides, not forget to my family who had given me a lot of support and encouragement. I thank to all my family especially to my husband Mohd Husairi bin Fadzilah Suhaimi and son, Muhammad Harith for their unrelenting love and patience towards me to achieve my final goal of obtaining a MSc. Not to forget my parents Siti Ragayah Omar and Zakaria Said. Also my sibling Mohd Zaileif, Shahrul Naim, Zaihasrol Hisam and Noraisah. I would like to thank all the technical support I received from Mr. Mohd. Azlan Jaafar, Ms. Nurul Wahida, Mr. Sailifairus Jaafar, and all other technical staff from preparation and characterization laboratory of Institute of Science, Faculty of Applied Sciences and Electrical, Mechanical and Chemical Engineering of UiTM. Lastly, I hope my project will help me to obtain knowledge and exposure in the field of science and technology as well as to introduce some basic techniques and tools that can be used in the future. A special note of gratitude goes to my fellow researchers, Ms. Che Rosmany, Ms Nurzahidatul Fathinah, Noor Asnida Asli, Hafsa Omar, Nur Amierah Asib, Noor Aadila Abd Aziz and other for their valuable assistance and discussions and all my fellow lab members and my close friends for their encouragements and assistance that kept me in high spirits throughout my journey towards achieving this research work. For those people around us who gives information and advice that related to our project. I am also grateful to Universiti Teknologi MARA and Ministry of Higher Education for the financial support given for this research work.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR’S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF ABBREVIATIONS	xiii
 CHAPTER ONE: INTRODUCTION	 1
1.1 Nanomaterial and Nanotechnology	1
1.2 Rare Earth Materials Lanthanum Phosphate	3
1.3 Problem Statements	4
1.4 Objective of Study	5
1.5 Scope and Limitation of Research	6
1.6 Organization of Thesis	7
 CHAPTER TWO: LITERATURE REVIEW	 8
2.1 Introduction	8
2.2 Rare Earth Materials	9
2.3 Synthesis and Deposition Lanthanum Phosphate	10
2.3.1 Introduction	10
2.3.2 Formation of Lanthanum Phosphate Nanostructures	11
2.3.3 Synthesis of Lanthanum Phosphate Nanostructures by Sol-gel Process	13
2.4 Properties of Lanthanum Phosphate	14
2.4.1 Structural Properties of Lanthanum Phosphate	14
2.4.2 Optical Properties of Lanthanum Phosphate	19
2.5 Potential Application of Lanthanum Phosphate	21